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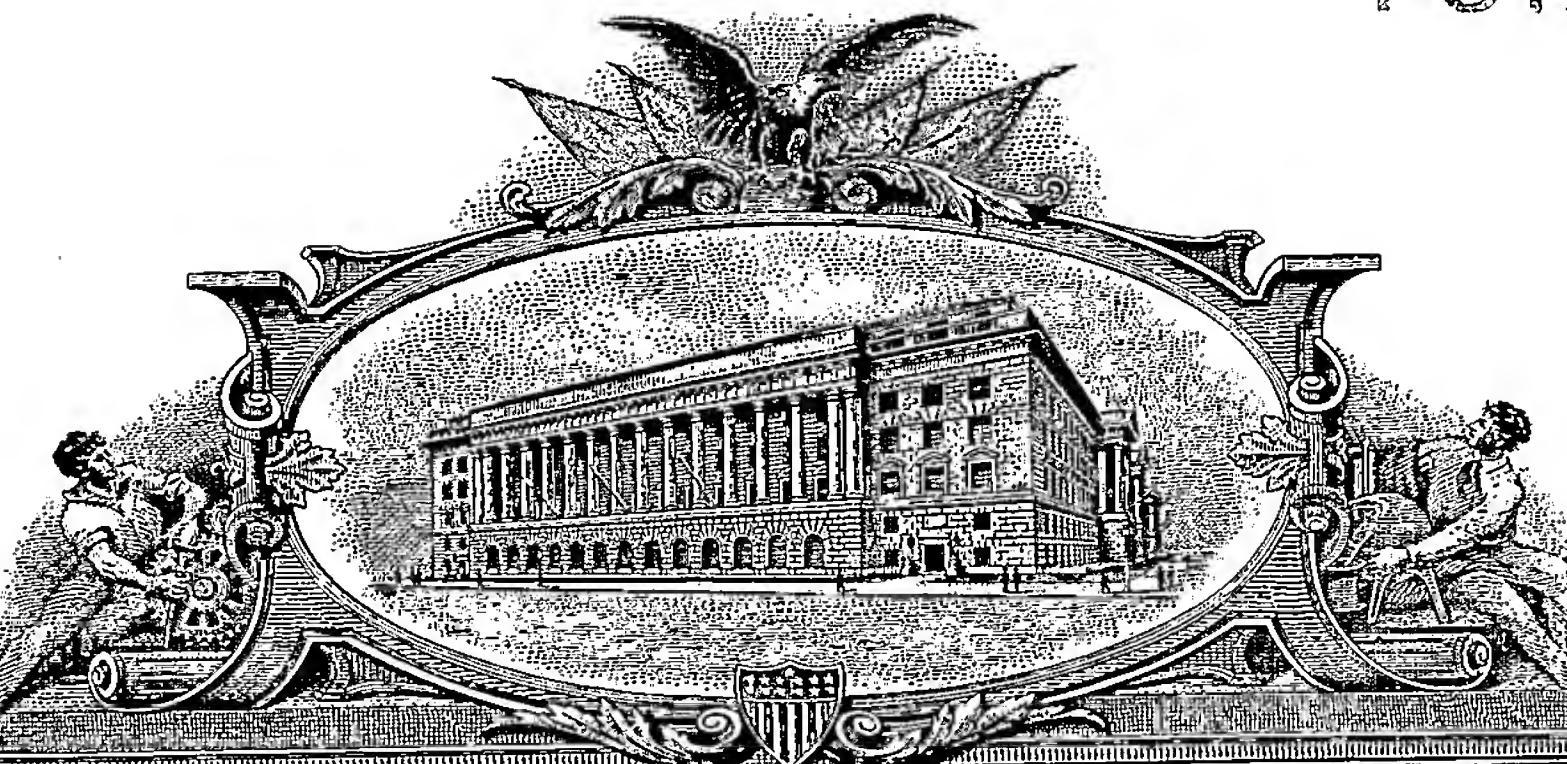
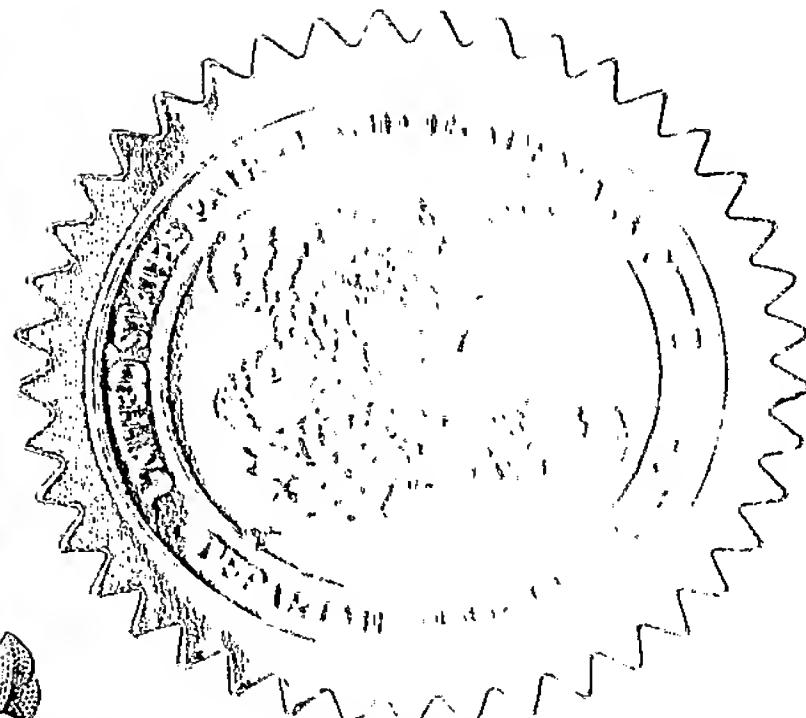
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Additional inventors are being named on the _____ separately numbered sheets attached hereto					
TITLE OF THE INVENTION (500 characters max)					
FLOW REVERSAL FOR PREVENTION OF PRECIPITATION FOULING IN PRESSURE-DRIVEN MEMBRANE PROCESSES					
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[Page 1 of 2]

Respectfully submitted

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Date DEC. 7, 2003

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Dec. 7, 2003

Gilron, Flow reversal for prevention of precipitation

Title: Flow reversal for prevention of precipitation fouling in pressure-driven membrane processes

Inventor: Jack L. Gilron

Invention Summary: A method to prevent fouling of a train of reverse osmosis or nanofiltration membrane elements by reversing the flow, such that the feed entrance becomes the concentrate exit and the concentrate exit becomes the feed entrance. This change is effected with a frequency such that the flow direction is changed before the induction time for precipitation corresponding to the composition of the supersaturated concentrate is reached. Thereby, precipitation fouling on the membrane is not allowed to commence. This allows running such membrane processes with higher recoveries, and/or less scale control chemicals. In the case of spiral wound elements it also prevents the buildup of biofouling deposits in the annular space between the spiral wound element and the pressure housing.

Background: Processes for water purification using membranes have become very widespread in general as evidenced in both trade and scientific articles and meeting reports. Two of the most widely used pressure-driven processes are reverse osmosis (RO) for removal of all salt species and nanofiltration (NF) with partial removal of monovalent salts and high removal of multi-valent salts. One of the main problems facing RO and NF is the occurrence of membrane fouling which leads to loss of production rate and loss of rejection or both (see [1] Water Treatment Membrane Processes, McGraw-Hill, 1996, ch 9). There are several kinds of fouling which occur:

- Colloidal fouling
- Organic fouling (adsorption of soluble organics on membrane surface)
- Biofouling – formation of a biofilm which by itself or in concert with other kinds of fouling causes deterioration of membrane performance
- Precipitation fouling (or scaling) due to precipitation of sparingly soluble salts

In the past a number of researchers [2-4] have suggested changing the flow direction in order to reduce concentration polarization and fouling in general. However none of them designed it to prevent precipitation fouling by sparingly soluble salts and one cannot learn from their work how to do so. The object of the present invention is primarily and precisely to mitigate this last kind of fouling but it has implications for biofouling as well. The acceptable methods to prevent precipitation fouling are to limit recovery so that concentrations of sparingly soluble salts do not greatly exceed saturation in the concentrate end. These saturation limits can be moderately increased by feeding antiscalants to the feed to the membrane which increase chemical pretreatment costs. In addition manufacturers require a minimum tangential flow rate in each commercial spiral element in order to minimize concentration polarization caused by the buildup of rejected salt convected to the membrane surface by the flux. With the new low pressure RO and NF membranes, that operate at pressures of 3-10 bar, axial pressure drops along the feed paths of the elements can significantly reduce the driving force for product water permeation so that flow rates must not be too high through the elements. These conflicting requirements make design of new plants quite difficult and a number of new strategies have been proposed [5].

According to crystallization theory, the time it takes for a supersaturated solution - in the absence of seed crystals or other appropriate pre-existing crystal growth surface -

to start to crystallize out its excess concentration is called the induction time. The induction time, τ , is related to the supersaturation, S , by the following equation:

$$\ln(\tau) = \frac{A}{[\ln(S)]^2} + B \quad (\text{eq. 1})$$

where S refers to the supersaturation ratio (actual conc. of the sparingly soluble salt in solution/saturation conc.). At high recoveries and high concentration polarization, the value of S at the membrane surface will be increasingly higher and the induction time will be increasingly lower (see for example [6]).

Description of the Invention

The aim of the present invention is to allow desalination membranes to operate under conditions where the composition of the solution next to the active membrane surface temporarily exceeds the effective saturation limit of sparingly soluble salts – while at the same time not allowing such local supersaturation to lead to precipitation fouling of the membrane. This is done in order to: relax design constraints, allow higher recoveries, and reduce brine volumes. Another aim of the invention is to provide a means of achieving very high recoveries despite temporarily high local supersaturations.

This invention includes a means and apparatus for reversing the flow in a train of RO or NF membrane elements in series. It is applicable to all geometries of membrane elements, including but not limited to spiral wound, hollow fiber (ID < 3mm), tubular (ID > 3 mm), plate and frame geometries familiar to the practitioner of membrane technology. It is also applicable to membrane elements made from all types of materials that can retain sparingly soluble salt species, including but not limited to polymerics (e.g. thin film composites of polyamide, polyureas, asymmetric membranes of cellulosic materials, sulfonated polysulfones, polymeric membranes with acid, base, oxidant and/or solvent stability) and ceramic materials (e.g. zirconium oxide, aluminum oxide, silica, carbon). It also includes a general methodology for changing the flow direction with a frequency that insures that the flow in one direction does not occur for a time exceeding the induction time corresponding to the composition of the high pressure solution at the concentrate end of the flow train.

As shown in equation 1, as the supersaturation increases the induction time to precipitation decreases. However when the supersaturation drops to 1, the induction time goes to infinity, in other words precipitation will not occur. The present invention is based on the hypothesis, herein proved, that when a membrane surface exposed to a supersaturated solution and is briefly exposed to an undersaturated solution, then the "clock of induction time is zeroed" as a result of such exposure to undersaturated solution. This means that when the membrane is again exposed to the supersaturated solution the induction time to precipitation begins to be measured from zero. Therefore if arrangements are made to change the solution next to the membrane to one whose composition is at saturation or less, at a time less than the induction time τ corresponding to the supersaturated solution, precipitation will never occur on the membrane because the induction time will never be reached. In order to practice this invention, one must know the induction time for the composition of greatest supersaturation that the membranes will encounter according to the design. This can be calculated from literature results relating induction times to solution

composition [see for example 6-8]. Alternatively this can be determined in a preliminary experiment on a small membrane unit.

The use of flow reversal to effect this periodic reduction in solution concentration is illustrated in figure 1a and 1b. In figure 1a, a feed stream (1), whose concentration of sparingly soluble salt is less than that of the saturation concentration ($C_1 < C_s$), is pressurized with a high pressure pump and the high pressure stream is fed to the left side (L) of a pressure vessel containing a series of one or more membrane elements (2). These membrane elements remove enough water through the membrane (M) into the permeate stream (3) that the exit stream (4) is concentrated in the retained sparingly soluble salts. The concentration of the sparingly soluble salts in the concentrate stream (4) is given by mass balance as :

$$C_4 = \frac{Q_1 C_1 - Q_3 C_3}{Q_4} \quad (\text{eq. 2})$$

where C refers to concentration (e.g. in g/L) and Q refers to volumetric flow rate (e.g. in L/hr). The subscript refers to the process stream to which the concentration and flow rate refers. Defining the recovery $Y = Q_3/Q_1$ as the portion of the feed stream (1) removed as permeate (3), one can rewrite equation 2 as:

$$C_4 = \frac{C_1 - YC_3}{(1 - Y)} \approx \frac{C_1}{(1 - Y)} \quad \text{when } C_3 \ll C_1 \quad (\text{eq 3})$$

If the recovery is high enough, then the sparingly soluble salt concentration at the membrane exit, C_4 , can be greater than the saturation concentration (C_s) and the membrane elements at the right end (R) of the pressure vessel sees a supersaturated concentrate solution. Because of concentration polarization, the concentration of sparingly soluble salt at the membrane wall (C_w) will be always be greater than the concentration (C) in the bulk solution. If conditions of concentrate flow (Q_4) are slow enough at the membrane element train exit (R), then the concentration polarization at the membrane wall can be so great that the concentration at the membrane wall (C_{wR}) can exceed C_s , even if the concentration of the bulk concentrate (C_4) is less than the saturation concentration (written mathematically as $C_{wR} > C_s > C_4$). Under conditions of supersaturation at the membrane surface, there will be an induction τ_{wR} associated with the composition C_{wR} . If the flow direction is then reversed (see figure 1b), then the membrane at the right end (R) of the pressure vessel (2) sees the feed which is at a concentration $C_1 < C_s$ and the time accumulated in the induction period is lost, and will only begin again when the situation is returned to the one obtaining in figure 1a. Similarly under the conditions described in figure 1b, if recovery is high enough and flowrate Q_4 is low enough, then the concentration (C_{wL}) next to the membrane at the left end (L) of the pressure vessel which is now the exit, will exceed the saturation concentration ($C_{wL} > C_s$) and this will be associated with an induction time, τ_{wL} . When the flow is reversed and the situation reverts to that illustrated in figure 1a, the time accumulated in the induction period at the left end (L) of the pressure vessel is also lost, and the induction time at the right end (R) must again begin at 0. To generalize this picture, the description describes not only the very ends of the pressure vessel but also conditions obtaining further in, where supersaturations can be still be reached. However the most extreme situations (and highest supersaturations will usually be obtained at the ends. In any event the claimed invention will be effective as long as every part of the membrane element train is

periodically exposed to a solution undersaturated with respect to sparingly soluble salts.

It could be argued that the same effect could be obtained by periodically reducing the pressure in the pressure housing (opening the back pressure valve downstream of the pressure vessels) without reversing flow. This method is described in the literature and is known as osmotic flushing. It has the distinct disadvantage of requiring loss of transmembrane pressure and loss of desalinated water production during the time of pressure reduction. Furthermore, periodic release and application of transmembrane pressure (often 5-20 bar) can result in the expansion and contraction of those elements that have a spiral wrapped configuration and this could lead to failure of the glue and other structural elements.

See figure 2 for a particular embodiment of how periodically reversing flow direction could be effected which is not intended to limit the general claim of flow reversal or the general approach of periodically reducing the concentration of sparingly soluble salts below their saturation limit. Figure 3 shows a laboratory setup for simulating the effect of flow reversal. See figure 4 for a typical trace of flux vs time showing the fouling induction time for calcium sulfate without flow reversal. Figure 5 shows the trace of flux vs time showing the prevention of fouling when a simulation of flow reversal was carried out.

Figure 2 shows the general layout of a particular embodiment of the system. It consists of the high pressure pump (HPP) the flow manifold consisting two 3-way valves (V2a, V2b) and one 2-way valve (V2c) and a back pressure valve for developing/maintaining pressure (V4) in the flow loop, a set of RO or NF membranes elements in series loaded in a pressure vessel (PV) and the appropriate ductwork and piping. In general, this pressure vessel (PV) can represent one or more actual pressure vessels in parallel all with the same number of membrane elements. In the standard direction the feed flow Q_1 is delivered to the left end of the pressure vessel and concentrate flow Q_4 exits from the right end of the pressure vessel. Under these conditions valve 2c is closed and valves 2a and 2b are positioned toward the down position. In switching to reverse flow (Q_1 enters PV from right and Q_4 exits the PV from the left), the following sequence of actions is taken: valve 2c is opened followed by moving V2b to up position followed by moving valve V2a to up position. This is done in this sequence to prevent causing a "water hammer" effect which could damage the membrane element. The unit is returned to standard flow direction by conducting the sequence in exact reverse of the first sequence.

In order to demonstrate the validity of the concept, a laboratory system was built to simulate what happens when a membrane element first sees the feed solution and then the concentrate solution. This unit is shown in figure 3. It consists of a flat sheet membrane test cell (TC) containing a flat sheet membrane sample (M), a high pressure pump (HPP) to pressurize the test fluid, flow meter (FI) to measure tangential flow rate in the test cell, pressure indicator (PI), a weigh scale (WS) to measure rate of permeation that is connected to a computer (PC), and two feed tanks (T1 and T2). Tanks T1 and T2 contain solutions that are undersaturated and supersaturated with calcium sulfate respectively. Two three way valves (V3a and V3b on the feed and return lines respectively) are used to switch operation between tanks T1 and T2. A fresh 5 micron filter was placed in line (FH) in each experiment

to remove suspended particulate so only calcium sulfate nuclei formed on the membrane could cause precipitation fouling and flux decline.

The effect of switching flow direction to prevent precipitation fouling is simulated by switching from tank T2 to tank T1 at time intervals that are less than the induction time for the supersaturated solution in tank T2. The experiments described below were conducted with a solution of 0.03 M CaSO₄ which is approximately 200% of the saturation concentration of pure calcium sulfate dehydrate (gypsum) in tank T2. This was formed by mixing equal volumes of 0.06 M H₂SO₄ and 0.06 M Ca(OH)₂. The tank T1 contained a solution of 0.01 M CaSO₄ which is undersaturated with respect to calcium sulfate dihydrate. In the blank experiment the solution was fed from tank T2 and the accumulated permeate was measured on the weigh scale (WS) and recorded on the computer (PC). From the weight change ΔW with time Δt the flux was calculated according to:

$$J = \frac{\Delta W}{\Delta t} \cdot \frac{1}{A} \quad (\text{eq. 4})$$

where A is the membrane area (34.2 cm²). From this the specific flux was calculated:

$$\text{Sp. Flux}(\text{kg/m}^2 \cdot \text{h} \cdot \text{bar}) = \frac{J}{\Delta P - \Delta \pi} \quad (\text{eq. 5})$$

where ΔP is the applied pressure $\Delta \pi$ is the difference between the osmotic pressure of the feed stream and the permeate stream. In the case of our study, the permeate stream osmotic pressure was negligible compared to the feed stream and $\Delta \pi$ was essentially equal to the osmotic pressure of the feed stream. In each experiment the specific flux was plotted as a function of time. If the specific flux began to drop this would be a sign that precipitation fouling had begun.

In an experiment using RO membrane BW30, figure 4 shows the results of running at a flux of 35 L/m²·h on a calcium sulfate solution at 200% of saturation concentration without switching the tanks with a feed flow rate of 100 L/hour (linear velocity of 0.73 m/s). After ~100 minutes, there is an initial dropoff in specific flux due to sealing the dead areas in the margins of the flow cell. After an additional plateau of another ~170 minutes, a steady decline in the specific flux of the membrane is commences. In this experiment, a mixer was used to keep the solution mixed in tank T2. In two more experiments, the same experiment was conducted without mixing the contents in tank T2, and in both experiments the induction time was found to be 60-65 minutes, i.e. steady decline in specific flux commenced after 60-65 minutes.

In a followup experiment with the same pressure and linear flowrate, same solution and the same membrane, the test feed solution was switched after 60 minutes from tank T2 to tank T1 containing 0.01 M calcium sulfate solution (~67% of saturation concentration) for ten minutes. Then the feed tank was again switched back to tank T2 with the supersaturated calcium sulfate solution for another hour. Conductivity results showed that the concentration in tank T2 did not change substantially during the experiment. This was repeated over an extended period of time. The results are shown in figure 5. The periodic disturbances in the calculated specific flux, represent the points at which the supersaturated solution was switched out for 10 minutes and replaced with undersaturated solution. As can be seen the flux is fairly stable for 360 minutes (6 hours). This shows the efficacy of the reverse flow method in preventing

precipitation fouling if the time between switches is kept lower than the induction time. This also provides evidence for the hypothesis that the induction time clock is zeroed in a system where the supersaturation condition is temporarily relieved.

Example:

This is a calculation example comparing performance of an RO unit desalting brackish water with calcium sulfate fouling potential using a standard tapered flow design and the example using reverse flow to reduce energy and chemical costs. This example uses the commercial membrane design software, Integrated Membrane Systems, of Hydranautics, Inc, but any standard commercial software could be used to generate the same results. Column 2 of table 1 represents the composition of the brackish water to be treated and is similar in its major ion composition to the water of wells sampled at Mashabe Sadeh in the Israel Negev region. The water is

Table 1: Compositions in RO of Negev region brackish water

Species	Raw	Feed	<u>Concentrate</u> (80% recovery)
Ca	206	206	973
Mg	72	72	402
Na	624	624	2632
K	17.5	17.5	78
NH4	0.3	0.3	1.3
CO ₃	0.3	0	0
HCO ₃	324	115.3	353
SO ₄	420	586.5	2714
Cl	1030	1030	4802
SiO ₂	17.5	17.5	74
General Properties			
pH	7.4	6	6.7
Temp C	25	25	25
TDS	2711.6	2672	12042
Conductivity	4713		
LSI	0.49	-1.36	0.51
%saturation (CaSO ₄)	11%	15%	102

Because the Langlier Saturation Index (LSI) (gap between actual pH and pH at which the given water would be at saturation with respect to calcium carbonate) is greater than 0, the water must be acidified if calcium carbonate is not to deposit. To effect this, acid must be added so that the feed pH is 6. As a result of adding 140 mg/L of sulfuric acid the treated feed water composition becomes as shown in column 3 of table 1. A standard design generating 20 m³/h of product at 80% recovery with 21 elements using low pressure brackish water RO membranes of type ESPA 1 (average flux of 26 L/m²-h) requires two stages in a 2-1 array with 7 elements in each of three pressure vessels. In order to keep the permeate flows balanced, a 4 bar booster pump is inserted between the 1st and second stage. After 80% recovery, the composition of the concentrate is given by column 4 in table 1. As can be seen the LSI is still slightly

positive (0.51) and the calcium sulfate concentration is slightly above saturation (102%). Since the concentration polarization is 1.07, the actual wall supersaturation (C_w/C_{sat}) is 109%. As a result, some antiscalant such as polyacrylates, polyphosphates or organophosphonates would be required to prevent scaling. The results of the conventional design are given in table 2 column 2.

An alternative design would be to place all three pressure vessels in parallel without a booster pump. This design would give a lower pressure drop resulting in less energy but the last element would be operated at less than the manufacturers minimum limit ($1.7 \text{ m}^3/\text{h}$ instead of $2.7 \text{ m}^3/\text{h}$). By reversing flow on the single stage, precipitation fouling would be prevented. The results for this are given in column 3 of table 2. It can be seen that the reverse flow design has the following savings: no antiscalant other than acid required, over 10% energy reduction, elimination of a booster pump of 1.5 kW (83% pump efficiency, 93% motor efficiency).

Table 2: Results of different designs for producing 20 m³/h at 80% recovery using ESPA1 8040 elements.

Design	Conventional with booster	Reverse flow
Stage 1		
No. Pressure vessels	2	3
No. of elements/PV	7	7
Inlet P, bar	8.6	9
Outlet P, bar	6.5	8.2
Permeate, m ³ /hr	14.5	20
Conc flow per PV, m ³ /h	5.25	1.7
Stage 2		
No. Pressure vessels	1	
No. of elements/PV		
Inlet P, bar	10.5	
Outlet P, bar	8.7	
Permeate, m ³ /hr	5.5	
Conc flow per PV, m ³ /h	5	
Average perm TDS (overall)	215	236
LSI (exit)	0.51	0.48
CaSO ₄ (% of sat) exit	102%	102%
CaSO ₄ (% of sat) exit wall	109%	116%
Energy consumption, kWh/m ³	0.46	0.405
Chemical consumption		
H ₂ SO ₄ (100%), g/m ³	140	140
Antiscalant, g/m ³	1	0

Claims

- 1. A general method for preventing precipitation fouling in a series of one or more pressure-driven membrane elements used for complete or partial desalination in which the pressurized flow is periodically reversed such that the feed is fed to the original concentrate exit and the concentrate leaves from the original feed entrance, such periodic switching being carried out at times less than the induction time corresponding to the highest supersaturation attained next to the membrane by supersaturated salts in the pressurized feed stream.**
- 2. Means for effecting the general method claimed in claim 1 including automated switching valves and appropriate flow manifolds.**
- 3. A method according to claim 1 in which the flow reversal is carried out within one or more stages of a pressure-driven desalination membrane system, where a stage is defined as a collection of one or more pressure vessels being operated in parallel.**
- 4. A method according to claim 1 effective in preventing precipitation fouling by one or more of the following sparingly soluble salts in their various mineral forms: calcium carbonate, calcium sulfate, silica, calcium phosphate, barium sulfate, strontium sulfate, calcium fluoride.**
- 5. Means according to claims 2 and 3 wherein two (2) three-way valves and one 2-way valve are used in such a way as elaborated in the patent description such that mechanical shocks of the type denoted as "water hammer" do not occur and the mechanical integrity of seals in the membrane element is not compromised.**
- 6. A method according to claims 1-5 wherein the membrane element is a spiral wound element.**
- 7. A method according to claims 1-5 wherein the membrane element is a hollow fiber (ID < 3 mm) element.**
- 8. A method according to claims 1-5 wherein the membrane elements are of a plate and frame geometry.**
- 9. A method according to claims 1-5 wherein the membrane elements are of a tubular (ID>3 mm) geometry.**
- 10. A method according to claims 1-6 allowing operation of membrane elements at pressurized feed flow rates less than those generally accepted by membrane element manufacturers in their conventional design software without suffering precipitation fouling, such that pressure drops and energy consumption are lower than that in conventional membrane design.**
- 11. A method according to claims 1-9 allowing operation of membrane elements with less antiscalant or acid chemicals than those generally accepted by manufacturers of antiscalant and or membrane elements manufacturers guidelines as reflected in their conventional design software without suffering precipitation fouling.**

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GILRON FRAKKESEN
Dec 7, 2003

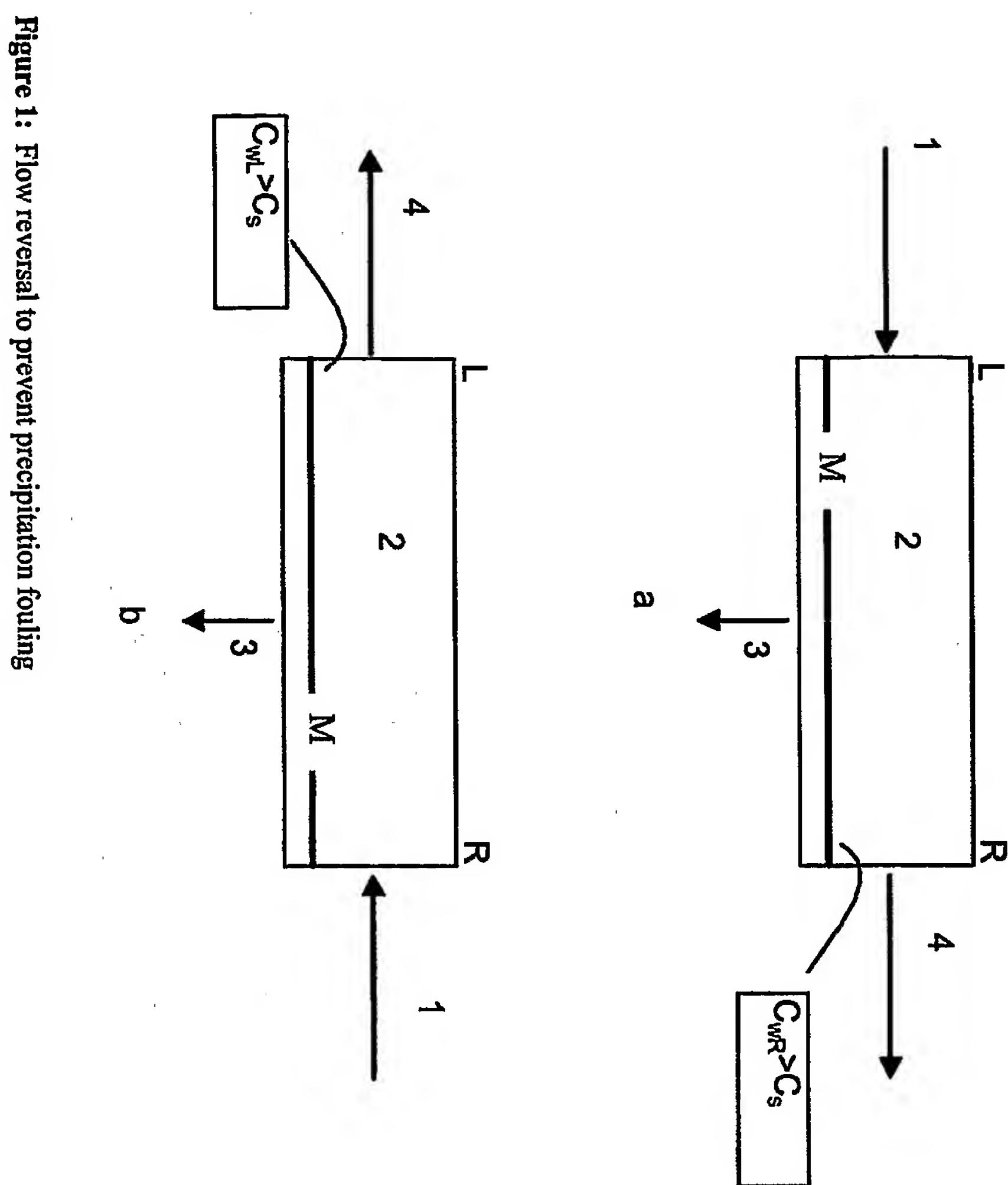


Figure 1: Flow reversal to prevent precipitation fouling

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Flow Reversal
Dec 7, 2003

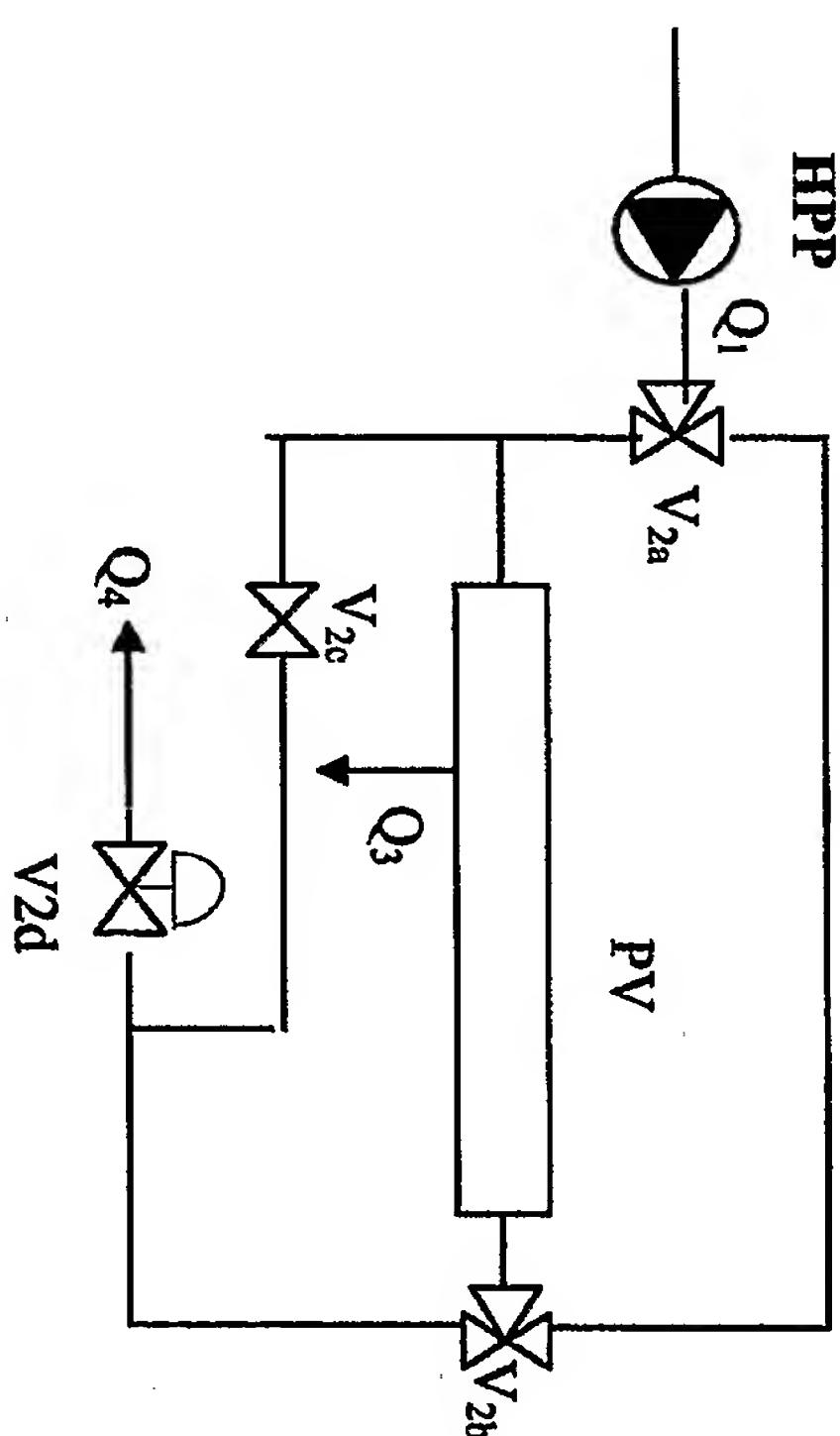


Figure 2: General arrangement for effecting flow reversal in a train of membrane elements loaded in a pressure vessel (PV).

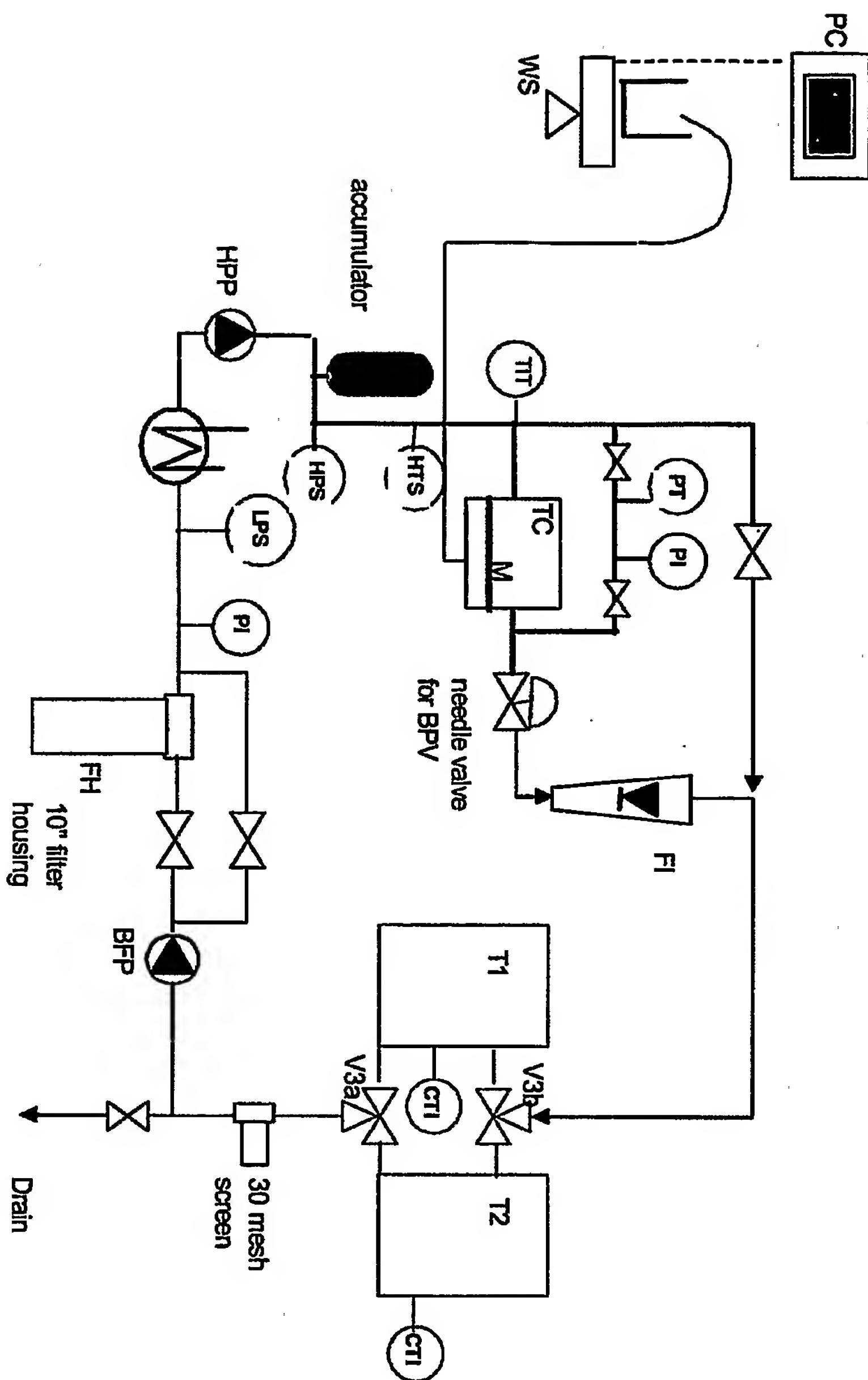


Figure 3: Laboratory setup for demonstrating reverse flow principle in preventing scaling

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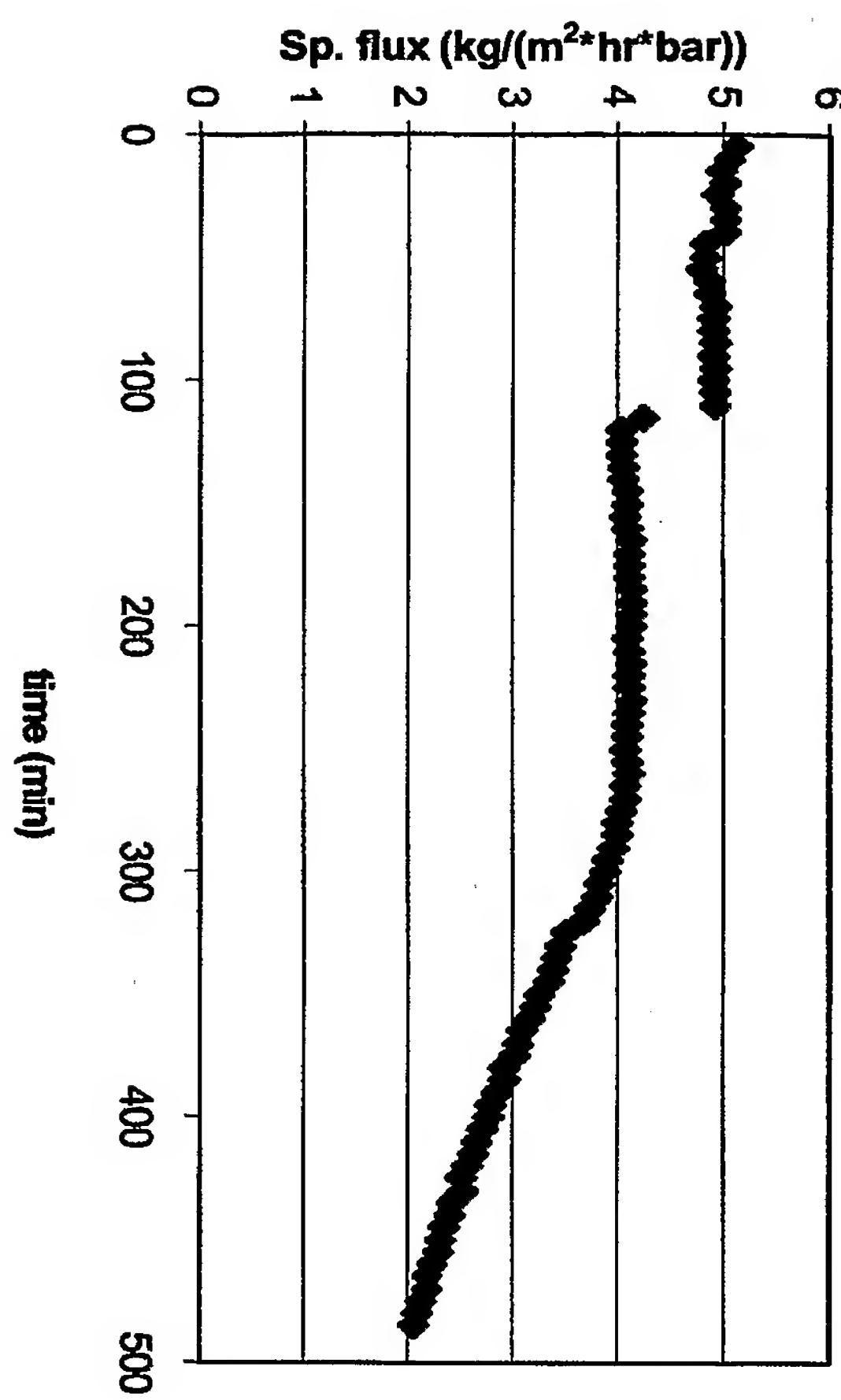


Figure 4: Experiment with 0.03 M CaSO₄ (200% supersaturation) without switching tanks.

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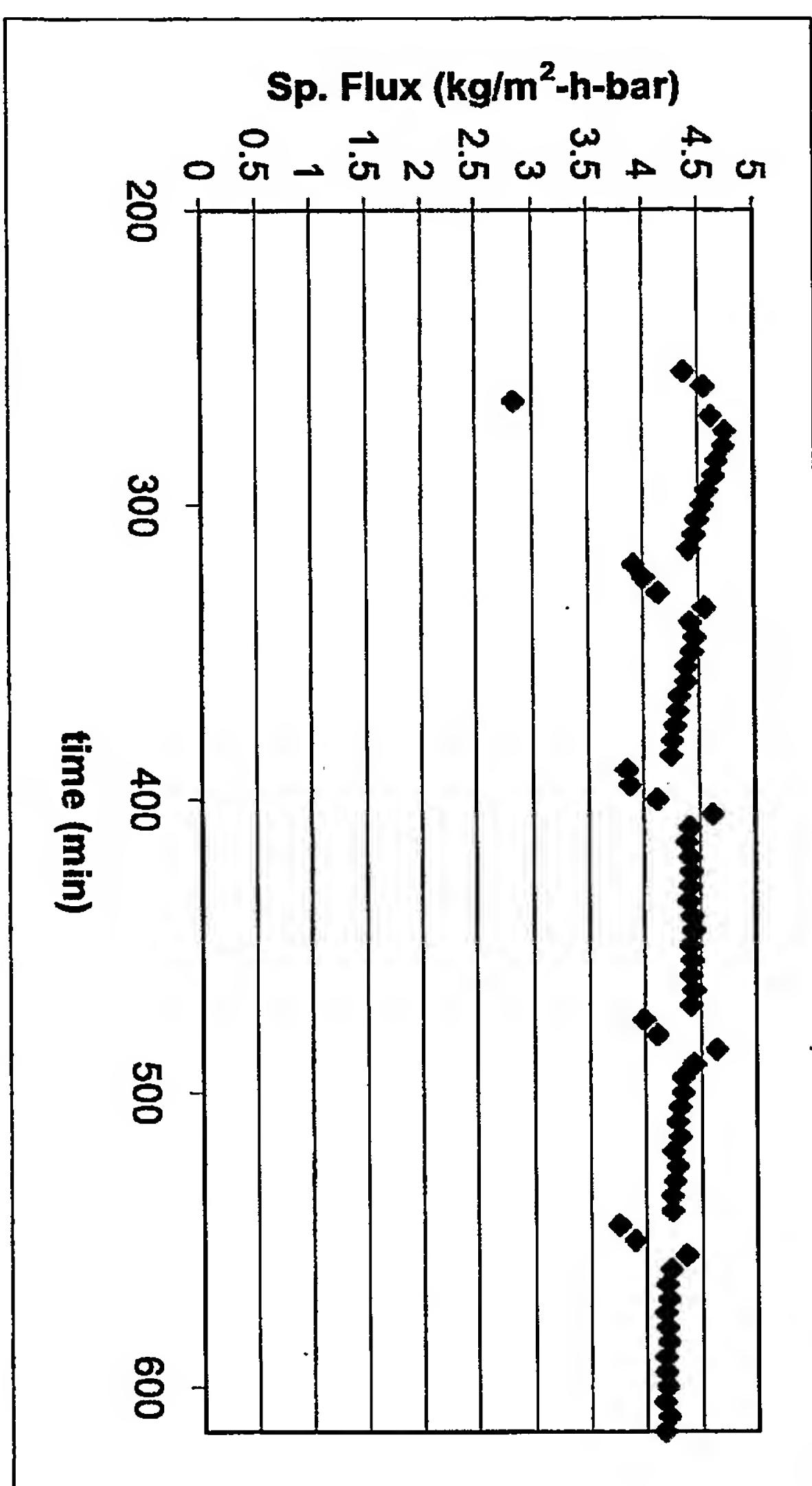


Figure 5: Experiment with 0.03 M CaSO₄ (200% saturation) switching to 0.01 M every 1 hour for 10 minutes.